Document made available under the Patent Cooperation Treaty (PCT)

International application number: PCT/US05/014884

International filing date: 29 April 2005 (29.04.2005)

Document type: Certified copy of priority document

Document details: Country/Office: US
Number: 60/566,972
Filing date: 30 April 2004 (30.04.2004)

Date of receipt at the International Bureau: 06 June 2005 (06.06.2005)

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)

World Intellectual Property Organization (WIPO) - Geneva, Switzerland
Organisation Mondiale de la Propriété Intellectuelle (OMPI) - Genève, Suisse

1325865

THE UNITED STATES OF AMERICA

TO ALL TO WHOM THESE PRESENTS SHALL COME:

UNITED STATES DEPARTMENT OF COMMERCE

United States Patent and Trademark Office

May 24, 2005

THIS IS TO CERTIFY THAT ANNEXED HERETO IS A TRUE COPY FROM THE RECORDS OF THE UNITED STATES PATENT AND TRADEMARK OFFICE OF THOSE PAPERS OF THE BELOW IDENTIFIED PATENT APPLICATION THAT MET THE REQUIREMENTS TO BE GRANTED A FILING DATE.

APPLICATION NUMBER: ***60/566,972***
FILING DATE: ***April 30, 2004***
RELATED PCT APPLICATION NUMBER: ***PCT/US05/14884***

UNITED STATES PATENT AND TRADEMARK OFFICE
DEPARTMENT OF COMMERCE

Certified by

Jon W. Dudas

Under Secretary of Commerce
for Intellectual Property
and Director of the United States
Patent and Trademark Office

Please type a plus sign (+) inside this box → +

PTO/SB/16 (5-03)
Approved for use through 04/30/2003. OMB 0651-0032
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

17589 U.S. PTO

17513 U.S. PTO 60/566972

043004

PROVISIONAL APPLICATION FOR PATENT COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c).

INVENTOR(S)

Given Name (first and middle [if any])	Family Name or Surname	Residence (City and either State or Foreign Country)
George K.	Strohmaier	Medina, OH
Nestor Daniel	Luchini	Naperville, IL
Eller D.	Frederiksen	Henderson, NV
Bob J.	Dull	O'Fallon, IL

☐ *Additional inventors are being named on the____ separately numbered sheets attached hereto*

TITLE OF THE INVENTION (280 characters max)

POLYUNSATURATED FATTY ACID DIVALENT METAL SALT SYNTHESIS

Direct all correspondence to: **CORRESPONDENCE ADDRESS**

☐ Customer Number ________ → *Place Customer Number Bar Code Label here*

OR *Type Customer Number here*

☒ Firm or Individual Name	Jimmie D. Johnson, Esq.				
Address	Synnestvedt & Lechner LLP				
Address	2600 Aramark Tower, 1101 Market Street				
City	Philadelphia	State	PA	ZIP	19107-2950
Country	USA	Telephone	215-923-4466	Fax	215-923-2189

ENCLOSED APPLICATION PARTS *(check all that apply)*

☒ Specification *Number of Pages* 37

☒ Drawing(s) *Number of Sheets* 2

☒ Application Data Sheet. See 37 CFR 1.76

☐ CD(s), Number ____

☒ Other (specify) Application Data Sheet, Abstract, Acknowledgment Postcard

METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT *(check one)*

☒ A check or money order is enclosed to cover the filing fees

☒ The Director is hereby authorized to charge filing fees or credit any overpayment to Deposit Account Number 19-5425

☐ Payment by credit card. Form PTO-2038 is attached.

FILING FEE AMOUNT ($) **$160.00**

The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.

☒ No.

☐ Yes, the name of the U.S. Government agency and the Government contract number are: ________

Respectfully submitted,

SIGNATURE [signature]

Date 4/30/2004

TYPED or PRINTED NAME Jimmie D. Johnson

REGISTRATION NO. 52,485
(if appropriate)
Docket Number: P22,888-F USA

TELEPHONE 215-923-4466

USE ONLY FOR FILING A PROVISIONAL APPLICATION FOR PATENT

CERTIFICATE OF MAILING BY "EXPRESS MAIL" (37 CFR 1.10) Applicant(s): **G. Strohmaier, N. Luchini, E. Frederiksen, B. Dull**			Docket No. **P22,888-F USA**
Serial No. **N/A**	Filing Date **Herewith**	Examiner **N/A**	Group Art Unit **N/A**
Invention: **POLYUNSATURATED FATTY ACID DIVALENT METAL SALT SYNTHESIS**			

I hereby certify that the following correspondence:

Provisional Application for Patent Cover Sheet, Patent Specification, Abstract, Application Data Sheet, Acknowledgment Postcard, Check for $160.00 for filing fee

(Identify type of correspondence)

is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 in an envelope addressed to: Mail Stop Provisional Application, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450 on

4-30-2004
(Date)

Christopher K. Ricco
(Typed or Printed Name of Person Mailing Correspondence)

(Signature of Person Mailing Correspondence)

EV143409679US
("Express Mail" Mailing Label Number)

Note: Each paper must have its own certificate of mailing.

P06A/REV02

POLYUNSATURATED FATTY ACID DIVALENT METAL SALT SYNTHESIS

CROSS-REFERENCE TO RELATED APPLICATION:

The present application is related to U.S. Patent Application Serial No. 10/716,292, filed November 18, 2003; U.S. Patent Application Serial No. 10/299,337, filed November 18, 2002, and U.S. Provisional Patent Application Serial No. 60/334,471 filed November 16, 2001. The disclosures of all three applications are incorporated by reference.

BACKGROUND:

The present invention relates to the preparation of unsaturated fatty acid divalent metal salt nutritional supplements from high glyceride content polyunsaturated oils, including marine oils. The present invention particularly relates to fatty acid divalent metal salts rich in desirable unsaturated fatty acids such as omega-3, omega-6, and omega-9 fatty acids, including eicosapentaenoic acid (EPA), docosahexaenoic acid (DHA), decosopentaenoic acid (DPA), linoleic acid (LN), arachidonic acid (AA), and linolenic acid (LA).

Certain divalent metals such as calcium, copper, iron, magnesium, manganese, selenium, and zinc have long been recognized as beneficial mineral nutrients for humans and certain companion animals and livestock, such as ruminants, horses, dogs, cats, rabbits, hamsters, birds, fish, and the like. For example, the mineral calcium not only builds and strengthens bones and teeth, it also maintains normal

heart beat and regulates blood pressure. It is also essential for the healthy functioning of the nervous system.

Copper is a key component of many enzyme systems. Copper deficiency is known to cause anemia, diarrhea, bone disorders, neonatal ataxia, changes in hair and wool pigmentation, infertility, cardiovascular disorders, impaired glucose and lipid metabolism and a depressed immune system.

Iron is involved in energy metabolism as an oxygen carrier in hemoglobin and as a structural component of cytochromes in electron transport. Iron is also a structural component at the catalytic site of a large number of enzymes covering a wide array of diverse metabolic functions including neurotransmitter synthesis and function, phagocyte antimicrobial activity, hepatic detoxification systems, and synthesis of DNA, collagen and bile acids.

As a positively charged divalent cation, magnesium acts as a calcium antagonist at the cell membrane level which is necessary to maintain normal electrical potentials and to coordinate muscle contraction-relaxation responses. Additionally, magnesium has roles in energy metabolism as a required cofactor for enzymes that catalyze fatty acid synthesis, protein synthesis, and glucose metabolism.

Manganese is a cofactor for enzymes involved in hydrolysis, phosphorylation, decarboxylation, and transamination. It also promotes activities of transferases such as glycosyltransferase, and of glutamine synthetase and superoxide dismutase. Manganese deficiency has been documented to cause poor growth and abnormal reproduction in animals.

Selenium is a component of glutathione peroxidases which are primarily responsible for reducing peroxide free radicals that include lipid peroxide formation

in cell membranes. Reduction of peroxides formed by oxidation of membrane phospholipids breaks the auto-oxidative chain reaction that damages cell membranes.

Zinc also is essential for protein synthesis, integrity of cell membranes, maintenance of DNA and RNA, tissue growth and repair, wound healing, taste acuity, prostaglandin production, bone mineralization, proper thyroid function, blood clotting and cognitive functions.

A variety of unsaturated fatty acids have been identified as desirable for producing a diversity of nutritional and physiological benefits in humans and lower animals, including companion animals and livestock, and accordingly have attracted attention as nutritional supplements. In certain animals, omega-3 fatty acids for example, have been discovered to promote fertility, promote health skin and coat, reduce inflammation, and have other nutritional and physiological properties as well. In humans, it is believed that omega-3 fatty acids such as EPA and DHA support healthy cardiovascular function, are important for visual and neuronal development, support healthy blood levels of cholesterol, triglycerides and very low density lipoproteins, and ease the inflammation associated with overuse of joints and improved carbohydrate metabolism. Conjugated Linoleic Acids (CLA's) have been discovered to possess a diverse and complex level of biological activity. Anti-carcinogenic properties have been well documented, as well as stimulation of the immune system. U.S. Pat. No. 5,914,346 discloses the use of CLA's to enhance natural killer lymphocyte function. U.S. Patent No. 5,430,066 describes the effect of CLA's in preventing weight loss and anorexia by immune system stimulation.

CLA's have also been found to exert a profound generalized effect on body composition, in particular, upon redirecting the partitioning of fat and lean tissue mass. U.S. Patent Nos. 5,554,646 and 6,020,378 disclose the use of CLA's for

reducing body fat and increasing lean body mass. U.S. Patent No. 5,814,663 discloses the use of CLA's to maintain an existing level of body fat or body weight in humans. U.S. Patent No. 6,034,132 discloses the use of CLA's to reduce body weight and treat obesity in humans. CLA's are also disclosed by U. S. Patent No. 5,804,210 to maintain or enhance bone mineral content.

It is also known that supplementing the diet of livestock with unsaturated fatty acids will alter the livestock fatty acid profile, so that, for example, feeding dairy cows and beef cattle a source of unsaturated fatty acids beneficial to humans will yield dairy and beef products for human consumption enriched with the beneficial unsaturated fatty acids. For example, U.S. Patent No. 5,143,737 discloses that the unsaturated fat content of milk and meat from ruminant animals can be increased by incorporating the intended unsaturated fat into the diet of the ruminant.

Thus, meat and milk enriched with CLA's and other unsaturated fatty acids can be obtained by supplementing ruminant diets with unsaturated fatty acids such as CLA. Dairy cows and beef cattle fed a source of CLA not only will produce lower fat content dairy and beef products, the products will be enriched with CLA's as well. Dietary supplementation of dairy cows and beef cattle with unsaturated fatty acids beneficial to humans can also be used to displace and thereby reduce the levels of undesirable saturated fatty acids in dairy and beef products.

The beneficial effects produced by unsaturated fatty acids are not limited to CLA's. Other unsaturated fatty acids are disclosed to be useful for treating diabetes (U.S. Patent No. 4,472,432), heart disease (U.S. Patent Nos. 4,495,201; 5,541,225 and 5,859,055), prostaglandin deficiencies (U.S. Patent No. 5,043,328), malaria (U.S. Patent No. 5,604,258), osteoporosis (U.S. Patent Nos. 5,618,558 and 5,888,541), cancer (U.S. Patent No. 5,763,484), immune system function (U.S. Patent No.

5,767,156), Huntington's Chorea (U.S. Patent No. 5,837,731) and inflammation (U.S. Patent No. 5,861,433). The disclosures of the foregoing patents are all incorporated by reference.

It has further been discovered that ruminants fed a source of trans-C18:1 fatty acids will have decreased concentrations of milk fat, hepatic triacylglycerol, and lower incidence of sub-clinical ketosis during early postpartum, and that feeding a source of linoleic (C18:2) fatty acids during the transition period will increase synthesis of $PGF_{2\alpha}$. The linoleic fatty acids thus hasten uterine involution and reduce the incidence of clinical and subclinical uterine inflammation; which translates to increased fertility.

Fatty acids are obtained through conversion of glycerides by either hydrolysis or saponification. Because of their fragile stability and complex degradation kinetics, certain unsaturated fatty acids, such as omega-3 fatty acids, have been difficult to incorporate into acceptable and effective nutritional supplements that are easily manufactured.

While divalent salts of saturated and unsaturated fatty acids have shown exceptional storage stability, unsaturated fatty acids typically do not readily react to form calcium salts using the processes known in the art such as those disclosed in U.S. Patent Nos. 5,143,737, 4,642,317; 4,826,694; 4,853,233 and 4,909,138. Instead of forming free-flowing granules, a mass develops that hardens into a tough material that resists grinding into the fine particles required for manufacturing nutritional supplements. The resulting material also lacks storage stability. The product tends to auto-oxidize through an exothermic reaction that leads to a congealing of the product mass from its free flowing granular state to a hard amorphous state, suggesting that significant quantities of unreacted starting materials are present in the final product.

U.S. Patent No. 6,576,667 discloses methods by which calcium salts of unsaturated fatty acids having as high as 60 percent by weight glycerides can be prepared. The disclosure of this patent is incorporated by reference. However, commercial sources of unsaturated fatty acids such as marine oils have glyceride contents as high as 100 percent by weight, which remain difficult to convert to storage stable free-flowing fatty acid divalent metal salts. U.S. Patent No. 6,576,667 addresses this problem by diluting the high glyceride content oils to glyceride levels below 60 weight percent with lower glyceride content fatty acid feedstocks such as Palm Fatty Acid Distillates (PFAD's). However, this also reduces the unsaturated fatty acid concentration in the fatty acid calcium salt product, requiring greater quantities to be fed as part of a feed ration to adequately supplement the ruminant diet.

U.S. Patent No. 6,229,031 discloses a saponification method by which calcium salts of fatty acids having as high as 100 percent by weight glycerides can be prepared. The disclosure of this patent is also incorporated by reference. However, calcium salts prepared from fatty acids with a significant degree of unsaturation prepared by this method have been discovered to lack storage stability.

A need exists for a method by which divalent metal salts of unsaturated fatty acids having acceptable storage stability can be prepared.

SUMMARY OF THE INVENTION:

This need is met by the present invention. It has now been discovered that improvements in the storage stability of saponified unsaturated fatty acid divalent metal salts can be significantly improved by performing the saponification method of U.S. Patent No. 6,229,031 in an atmosphere in which the partial pressure of oxygen has been reduced. While it is known to exclude oxygen from a reaction process to prevent explosion or fire, or the oxidation of the ingredients and end product at elevated temperatures during the course of a reaction, what was unexpected is that minimizing atmospheric oxygen in the method of the present invention also improves the shelf life and storage stability of the finished product long after the reaction is completed.

The shelf life and storage stability of unsaturated fatty acid divalent metal salt products varies with unsaturated fatty acid content and degree of unsaturation. Shelf life decreases as polyunsaturated fatty acid content increases. Unsaturated fatty acid divalent metal salt products must possess sufficient shelf life and storage stability to pass without objection in the field. For purposes of the present invention, "shelf life" is defined as the time period required before product degradation exceeds levels considered acceptable to those of ordinary skill in the art. For a product to have adequate storage stability, the shelf life must exceed the amount of time the product would be expected to spend in storage after manufacture and before being consumed. Typically this is about six months.

High levels of mono-unsaturated fatty acids in glyceride sources are needed before a saponified divalent metal salt product will exhibit storage instability over a typical shelf life. Significantly low quantities of polyunsaturated fatty acids, will reduce product shelf life to unacceptable levels. At lower mono-unsaturated fatty acid

concentrations the presence of polyunsaturated fatty acids will result in storage instability in an otherwise stable mono-unsaturated fatty acid divalent metal salt product with an adequate shelf life. Nevertheless, many fatty acid glyceride sources have mono- and polyunsaturated fatty acid levels that will produce a storage stable divalent metal salt product. Palm oil calcium salts, for example, possess adequate storage stability when prepared by the method of U.S. Patent No. 6,229,031.

The method of the present invention is thus particularly useful when used to saponify fatty acid glycerides with levels of mono- and polyunsaturated fatty acids that would otherwise produce an unstable divalent metal salt product when saponified in the presence of oxygen. For purposes of the present invention, such fatty acid glycerides are fatty acid feedstocks with an unsaturated fatty acid concentration sufficient to form unstable divalent metal salt products when saponified in an ambient atmosphere, wherein unstable divalent metal salt products are defined as divalent metal salt products with an inadequate "shelf life" as that term is defined in the present specification.

Therefore, one aspect of the present invention is a method for the preparation of a free-flowing, storage-stable fatty acid divalent metal salt product by forming a reactive admixture of (a) an unsaturated fatty acid glyceride feedstock and (b) from about 10% to about 30% of the total admixture weight of divalent metal hydroxide, and then heating the admixture to a temperature at which the fatty acid glycerides saponify to form fatty acid divalent metal salts in an atmosphere in which the partial pressure of oxygen has been reduced by an amount effective to provide an improvement in storage stability, until a free-flowing, storage-stable product is obtained; wherein the divalent metal is selected from calcium, copper, iron, magnesium, manganese, selenium, and zinc.

The partial pressure of oxygen in the reactive atmosphere may be reduced by inert gas blanketing of the admixture with, for example, nitrogen, carbon dioxide or argon, or by heating the admixture under vacuum. Nitrogen blanketing methods are preferred, as are methods in which atmospheric oxygen is essentially eliminated.

The saponification method of the present invention is typically applied to fatty acid glyceride feedstocks having greater than about 45% by weight of the fatty acid content in the form of fatty acid glycerides, and having an unsaturated fatty acid concentration sufficient to form unstable divalent metal salt products when saponified in an ambient atmosphere. The method can be used to prepare storage-stable products from unsaturated fatty acid sources such as marine oils having glyceride levels as high as 100% by weight.

According to another aspect of the present invention storage-stable unsaturated fatty acid divalent metal salt products are provided that are prepared by the method of the present invention. Such products can assume the form of powders, granules, paste, pellets, emulsion, colloidal suspension, non-colloidal suspension, capsules, and tablets, and may be administered orally, enterally, rectally, topically, or nasally. Moreover, such products may also comprise vitamins, antioxidants, amino acids, sugars and complex carbohydrates, trace nutritional elements, medicaments, proteins, anabolic steroids, hormones related to pregnancy or lactation, herbal supplements, lactobacillus micro-organisms, and cosmetological active ingredients.

The present invention thus provides storage-stable divalent metal salts of unsaturated fatty acids beneficial to humans, companion animals, and livestock from feedstocks for those acids that are typically very high in glyceride content, without having to significantly dilute the feedstock, if at all, with low glyceride content feedstocks that contain little, if any, of the beneficial unsaturated fatty acids. Thus,

essentially any unsaturated oil containing useful levels of beneficial unsaturated fatty acids is suitable for use with the present invention, and for purposes of the present invention is included within the definition of an unsaturated oil.

The present invention can be used with unsaturated oils having what was until now considered a low level of beneficial unsaturated fatty acids among the total unsaturated fat content, because with the present invention there is no need to dilute the feedstock with saturated fatty acids and the unsaturated fatty acid concentration is essentially conserved over time. Storage-stable divalent metal salts of unsaturated oils containing between about 3 and about 100 weight percent unsaturated fatty acids based on the total fatty acid content can be formed using the methods of the present invention. Oils with lower levels of beneficial unsaturated fatty acids may be used if they have utility based on their beneficial unsaturated fatty acid content.

Among the beneficial unsaturated fatty acids, beneficial polyunsaturated fatty acids are particularly preferred, and especially omega-3 fatty acids such as EPA, DHA, DPA and LNA, and omega-6 fatty acids such as linoleic acid, CLA's and arachidonic acid, because of their reproductive and other benefits. Oils which are a high glyceride content source of omega-3 and omega-6 fatty acids, such as marine oils, may be directly saponified by the method of the present invention to form storage-stable divalent metal salt products without first diluting the oil with saturated fatty acids. The resulting products contain storage-stable levels of omega-3 and omega-6 fatty acid divalent metal salts that heretofore could not be attained using prior art methods.

Therefore, according to yet another aspect of the present invention a free-flowing storage-stable fatty acid divalent metal salt product is provided containing at least one unsaturated fatty acid, wherein the total unsaturated fatty acid content is

between about 40 and about 95% by weight. Preferred products contain beneficial polyunsaturated fatty acids such as omega-3 and omega-6 fatty acids, with a product containing at least one polyunsaturated fatty acid selected from DHA, EPA, DPA, LNA, linoleic and arachidon-ic acid, each at a level between about 1 and up to about 80% by weight being particularly preferred. Conjugated polyunsaturated fatty acids such as CLA's are also preferred.

The DHA-and EPA- containing fatty acid divalent metal salts enhance the fertility of ruminants and other animals, including humans. Therefore, according to still yet another aspect of the present invention, a method is provided for increasing fertility in a animal, in which the animal is fed an effective amount of the EPA- and DHA- containing fatty acid divalent metal salts of the present invention.

The method according to this aspect of the present invention is particularly effective to enhance the fertility of ruminants, especially dairy cows. Methods in accordance with this aspect of the present invention begin feeding the supplements daily to a female ruminant from about 21 days before to about 28 days after parturition and feeding continue at least until conception occurs.

The fertility enhancement obtained by the calcium salts of the present invention also includes a reduction in embryonic death in the months following conception. Therefore, methods in accordance with the present invention continue feeding the supplements for at least 30 days, and preferably for at least 60 days after conception.

The foregoing and other objects, features and advantages of the present invention are more readily apparent from the detailed description of the preferred embodiments set forth below, taken in conjunction with the accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 depicts a batch process according to one embodiment of the present invention;

FIG. 2 depicts a continuous process according to another embodiment of the present invention;

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS:

The present invention provides a process by which unsaturated marine, animal, and vegetable fats, oils and other unsaturated fatty acid glyceride content materials may be converted to a storage-stable fatty acid divalent metal salts having utility as nutritional supplements for humans and animals. By divalent metal salt is it meant a salt of any biocompatible metal having a valence of two, such as calcium, copper, iron, magnesium, manganese, selenium, and zinc. By biocompatible it is meant having the property of being biologically compatible by not producing a toxic, injurious, or immunological response in living tissue. These salts may be in the form of free-flowing powders, granules, paste, or pellets, or may be in the form of an emulsion, colloidal suspension, non-colloidal suspension, capsules, or tablets.

Typical fatty acid feedstocks range in fatty acid content between C_{10} and C_{22} fatty acids and fatty acid glycerides. The term "glyceride" as employed herein includes C_{10}-C_{22} fatty acid monoglycerides, diglycerides and triglycerides, and any mixture thereof. The economic advantages provided by the present invention are obtained when using fatty acid feedstocks having glyceride concentrations that prevent the formation of stable divalent metal salt products or when using fatty acid feedstocks with unsaturated fatty acid concentrations that prevent the formation of stable divalent metal salts through high temperature saponification in an ambient atmosphere. Usually, fatty acid feedstocks cannot be hydrolyzed and neutralized to form stable divalent metal salts at glyveride contents of about 45% by weight and greater. However, the method of the present invention can be employed with fatty acid feedstocks in which from about 15% to about 100% by weight of the fatty acids are in glyceride form. The method of the present invention will also form divalent

metal salts using lower glyceride content fatty acid feedstocks, and with glyceride-free fatty acid feedstocks.

The method of the present invention will improve the storage stability of essentially any saponified unsaturated fatty acid divalent metal salt product, even those considered by the ordinarily skilled artisan to be adequately storage stable. When the level of polyunsaturated fatty acids is less than about 20% by weight, significant improvement in divalent metal salt product stability is obtained for fatty acid feedstocks with total unsaturated fatty acid concentrations greater than about 50% by weight. When the level of polyunsaturated fatty acids is between about 20 and about 90% by weight, significant improvement in divalent salt product stability is obtained for fatty acid feedstocks with total unsaturated fatty acid concentrations greater than about 25% by weight.

Thus, the process of the present invention can be used to prepare fatty acid divalent metal salts from pure unsaturated oils of marine, animal, or vegetable origin, including those disclosed in the above-referenced U.S. Patent Number 6,576,667. Examples of suitable vegetable oils include soybean oil, cottonseed oil, linseed oil, canola oil, and the like, and oils derived from marine vegetation such as algae, kelp, and the like. Examples of marine oil sources include menhaden, herring, mackerel, caplin, tilapia, tuna, sardine, pacific saury, krill, salmon, anchovy, skates, whale, seal, crab, shrimp, lobster, eel, mollusk, and the like.

Such fatty acid glyceride feedstocks typically contain from about 10 to about 100 weight% of the fatty acid content in the form of fatty acid glycerides, from about 0 to about 90% by weight of free fatty acids, and less than 5% by weight of moisture, insolubles and unsaponifiables. The feedstocks also typically contain from about 10 to about 95% by weight, and preferably between about 20 and about 90% by weight,

of total unsaturated fatty acids, with between about 15 and about 80% by weight of the total unsaturated fatty acid content being polyunsaturated. The method of the present invention thus provides storage stable fatty acid divalent metal salts containing between about 15 and about 85% by weight of one or more unsaturated fatty acids based on total product weight, of which between about 10 and about 80% by weight are one or more polyunsaturated fatty acids. The divalent metal salts contain less than 10% by weight, and preferably less than 1% by weight of unreacted glycerides.

Other conventional biologically active materials can be added to the divalent metal salt products at conventional levels by known means. By the term "biologically active material", it is meant any substance capable of being administered to a living organism that produces a physiological or biochemical effect on one or more vital processes occurring in that living organism. The biologically active material can be selected from a broad variety of nutrients and medicaments, either as a single component or as a mixture of components, which are illustrated by the following list of active molecular species:

1. Sugars and complex carbohydrates which include both water-soluble and water-insoluble monosaccharides, disaccharides, and polysaccharides. Particularly preferred carbohydrates include cane molasses and sugar beet byproducts.

2. Amino acid ingredients, either singly or in combination, which include arginine, histidine, isoleucine, leucine, lysine, methionine, phenylalanine, threonine, tryptophan, valine, tyrosine ethyl HCl, alanine, aspartic acid, glutamic acid, sodium glutamate, potassium glutamate, glycine, proline, serine, cystine ethyl HCl, and the like; and analogues and salts thereof.

3. Vitamin ingredients, either singly or in combination, including thiamine HCl, riboflavin, pyridoxine HCl, niacin, inositol, choline chloride, calcium pantothenate, biotin, folic acid, ascorbic acid, p-aminobenzoic acid, cobalamin, n-dimethyglycine, calcium pangamate, choline bitartrate, thiamin, niacin, pantethine, beta-carotene, lutein, lycopene, chondroitin, glucosamine, d-alpha tocopherol, calciferol, and derivatives of 2-methyl-1,4-naphthoquinone, and the like.

4. Trace element ingredients, either singly or in combination, including compounds of cobalt, tin, nickel, chromium, molybdenum, iodine, chlorine, silicon, vanadium, iodine, sodium and potassium.

5. Protein ingredients as obtained from sources such as cottonseed meal, soybean meal, canola meal, sunflower seed meal, canola meal, safflower meal, dehydrated alfalfa, corn gluten meal, soybean protein concentrate and potato protein, marine meal, marine and poultry protein isolates, crab protein concentrate, hydrolyzed protein feather meal, poultry byproduct meal, liquid or powdered egg, milk whey, egg albumen, casein, marine solubles, cell cream, brewer's residues, and the like.

6. Medicament ingredients, either singly or in combination, including promazine hydrochloride, chloromedoniate acetate, chlorotetracycline, sulfamethazine, monensin, sodium monensin, poloxaline, oxytetracycline, BOVATEC, streptomycin, and the like.

7. Antioxidants, including butylated hydroxyanisole, butylated hydroxytoluene, tertiary-butylhydroquinone, tocopherols, propyl gallate and ethoxyquin; and preservatives, including sodium sorbate, potassium sorbate, sodium benzoate, propionic acid, .alpha.-hydroxybuteric acid, and the like.

8. Herbal supplements, including St. John's Wart, echinacea, Goldenseal, and ginkgo, aloe, astragalus, black cohosh root, sarsaparilla root, Siberian ginseng root,

licorice root, blessed thistle herb, squaw vine herb and false unicorn root. bladderwrack, cascara sagrada, chamomile, chickweed, damiana leaves, devil's claw, dong quai, hoelen, ophiopogon, scute, platycodon, citrus morus root, fritillary, gardenia, shizandra, Camellia sinensis (green tea), and Lavandula officinalis, Carthamus tinctorius, Simmondsia chinensis, Sesamum indicum, Vitis vinifera, Persea gratissima, cucumis sativus, organic Macrocystis pynfera, and the like.

9. Lactobacillus micro-organisms, including Acidophilus, Bifidus, and Rhamnosus, and the like.

10. Cosmetological active ingredients, including bisabolol, phytantriol, retinol, benzophenone-1, and the like.

11. Hormones involved in pregnancy and lactation, including chorionic gonadotrophin, progesterone, estrogen, fetal adrenal axis hormones, relaxin, prostaglandins, prolactin, oxytocin, and the like.

12. Anabolic steroids including tetrahydrogestrinone, trenbolone, gestrinone, and the like.

While the calcium salts of the present invention can be used as rumen-inert feed supplements for ruminants such as cattle, these and other unsaturated fatty acid divalent metal salts are also useful in general as a nutritional supplement for humans and other mammals, including pets such as dogs and cats, and non-mammals, including birds and fishes, when formulated to contain unsaturated fatty acids beneficial to the respective species. The beneficial unsaturated fatty acid calcium salt nutritional supplements can also be fed to livestock to produce meat, poultry and dairy products enriched with the beneficial unsaturated fatty acids for consumption by species for which the unsaturated fatty acids are beneficial.

Examples of specific unsaturated fatty acids that are beneficial to humans, livestock, pets, birds or fishes, and which can be converted by the saponification method of the present invention to storage-stable fatty acid divalent metal salts, include linoleic acid (C18:2), arachidonic acid (C20:4) and isomers thereof, omega-3 fatty acids such as DHA, EPA, DPA, LNA, and the like, omega-6 fatty acids, CLA isomers having utility as human dietary supplements, including the 10,12 and 9,11 isomers, specific examples of which include the trans 10, trans 12; trans 10, cis 12; cis 10, trans 12; cis 10, cis 12; trans 9, trans 11; trans 9, cis 11; cis 9, trans 11 and cis 9, cis 11 isomers, trans fatty acids isomers having utility as dietary supplements for livestock, including C18:1 isomers such as trans-9-octadecenoic acid. A particularly preferred product contains at least one polyunsaturated fatty acid selected from, about 1 to about 25% by weight DHA, about 1 to about 25% by weight EPA, about 1 to about 25% by weight DPA, about 1 to about 75% by weight LNA, about 0.5 to about 10 % by weight arachidonic acid, about 1 to about 80% by weight linoleic acid and about 1 to about 80% by weight CLA.

The fatty acid divalent metal salts are prepared by adding a divalent metal hydroxide, such as $Ca(OH)_2$, $Cu(OH)_2$, $Fe(OH)_2$, $Mg(OH)_2$, $Mn(OH)_2$, $Se(OH)_2$, and $Zn(OH)_2$, to the fatty acid glyceride-containing feedstock in the range of from about 10 to about 30% by weight of the total composition. A divalent metal hydroxide level between about 12 and about 18% by weight of the total final divalent metal salt product composition is preferred.

Additional heat is added to the admixture, if necessary, to increase the temperature to a range between about 150 and about 300° C., and preferably between about 200 and about 270°C. In accordance with the present invention, the divalent metal hydroxide may be substituted by a stoichiometric equivalent of divalent metal

oxide (such as CaO, CuO, FeO, MgO, MnO, SeO, and ZnO) and water, and for purposes of the present invention the term "divalent metal hydroxide" in the claims is defined as including the replacement in the reactive admixture of divalent metal hydroxide by a stoichiometric quantity of divalent metal oxide and water.

The reaction can be performed under vacuum, ambient pressure or at an elevated pressure to maintain the desired temperature. The reaction is performed in an atmospheric environment in which the partial pressure of oxygen is reduced to levels at which a storage-stable saponified divalent metal salt product will form. Oxygen level reduction may be achieved either by purging a sealed reaction vessel with an inert gas such as nitrogen, carbon dioxide or argon, or by drawing a vacuum. Methods for inert gas purging and vacuum drawing are essentially conventional and well known to those skilled in the art. For example, inert gas purging can be accomplished using an inert gas blanket consisting of, for example, nitrogen, carbon dioxide or argon, at a flow rate between about 0.25 to about 50 liters per minute, and preferably between about 1.0 to about 20.0 liters per minute that is applied to the reaction vessel. A vacuum of between about 250 to about 750mm Hg, and preferably between about 300 to about 500mm Hg, should be drawn on the sealed vessel, and preferably using an inert gas such as nitrogen, carbon dioxide or argon as the vacuum gas.

Any reduction of the partial pressure of oxygen in the reactive atmosphere will provide an improvement in storage stability. The reduction needed to create a storage-stable product with an adequate shelf life will depend upon the level and degree of unsaturation in the fatty acid glycerides to be saponified, with higher levels and degrees of unsaturation requiring greater reduction in the oxygen content of the reactive atmosphere. Typically the partial pressure of oxygen should be less than

about 100 torr, with partial pressures less that 50 torr preferred and partial pressures less that 10 torr even more preferred. The reduction in the partial pressure of oxygen is attained either by displacement of oxygen with the inert gas purge or by drawing a vacuum sufficient to attain a sufficient reduction in total pressure to reduce the partial pressure of oxygen to desired levels, or both.

The amount of time required for the saponification reaction is typically between about 1 to about 15 minutes, and more typically between about 2 and about 5 minutes. The stability of the divalent metal salts is improved by limiting the reaction time. The reaction is easily identified by the transformation of the admixture into a caramel-like mass. Upon further heating and agitating, the mass further transforms into a taffy-like material, which, upon transfer from the reaction vessel, can easily be processed into free-flowing particles.

In an alternate embodiment, the divalent metal salt stability is improved by method steps that accelerate the reaction process and thereby limit the total quantity of heat to which the reactive admixture is exposed. According to this particular embodiment, the fatty acid feedstock is preheated to a temperature between about 350 and 550°F and then rapidly combined with the divalent metal salt at a rate effective to form a uniform, homogenous mass having improved storage stability over products formed without preheating and rapid blending, typically within a minute. Preferred mixing rates will form a uniform homogenous mass within one to 15 seconds. Adequate heat is supplied to the feedstock until a free-flowing storage-stable product is obtained.

The process of the present invention may be employed as either a batch or a continuous process. Examples of reaction vessels suitable for use with the present

invention include continuous or batch reactors, indirectly or directly heated, with multiple agitation and shear elements, suitable for very high viscosity materials.

A batch process according to the present invention is depicted in FIG. 1. Fatty acid glycerides and a divalent metal hydroxide are added via lines 16 and 17, respectively, to the interior 18 of sealed production vessel 10 adapted to supply heat to the vessel contents (not shown), as well as to remove heat therefrom (not shown). The production vessel is equipped with blades 12 for mixing the vessel contents under adequate shear to form a homogenous admixture of the vessel contents.

The production vessel should also be adapted to remove oxygen from the reaction environment, either by forming a vacuum above the vessel contents, or by blanketing the vessel contents with an inert gas such as nitrogen, carbon dioxide or argon. Means by which this can be accomplished are well known to the artisan of ordinary skill. In the embodiment of FIG. 1, line 20 supplies a purge of inert gas to the sealed vessel, in this case nitrogen, which is vented via line 21. Line 21 can be readily be adapted to draw a vacuum as well.

The reaction mixture is heated, reacted and cooled under either the inert gas purge and/or vacuum. After cooling, the mixer blades 12 grind the product into flakes and granules that are discharged from the rector at port 22.

A continuous process according to the present invention is shown in FIG. 2. Feedstock oils are supplied via line 50 to heater 52. Divalent metal hydroxide is supplied via line 54 and combined with the heated feedstock oil, which is supplied from the heater via line 56. The combination of divalent metal hydroxide and feedstock oil is then supplied via line 58 to reaction screw conveyor 60, the screw flight 62 of which functions to mix the contents under adequate shear to form a

homogenous admixture thereof. The screw conveyor is adapted to supply heat to the reaction mixture (not shown).

Line 64 supplies a purge of inert gas, which in this case is also nitrogen, that exits via vent 66. These lines can also be adapted to draw a vacuum. The product is discharged via line 68 to cooling and grinding screw conveyor 70, the screw flight 72 of which functions to grind the product into flakes and granules that are discharged from screw conveyor 70 at port 74. Screw conveyor 70 is adapted to withdraw heat from the reaction mixture (not shown). Line 76 supplies a purge of inert gas, which is also nitrogen, that exits via vent 78. These lines can also be adapted to draw a vacuum.

The free-flowing particles produced by the aforementioned process can subsequently be formulated into products of various forms including powders, granules, pastes, pellets, emulsions, colloidal suspensions, non-colloidal suspensions, elixirs, capsules, or tablets to be administered through a variety of techniques known in the art including, but not limited to, orally, enterally, rectally, topically, parenterally, and nasally. Depending on the dosage form, it may be necessary to grind or mill the particles to obtain a particle size suitable for the manufacture thereof. Particle size requirement are well known to those skilled in the art of manufacturing products of this type.

Product compositions according to this invention may be prepared according to the customary methods, using one or more acceptable adjuvants or excipients. The adjuvants comprise, inter alia, diluents, sterile aqueous media, and various non-toxic organic solvents. The compositions may comprise sweeteners such as sucrose, lactose, fructose, saccharin, or Nutrasweet; flavorings such as peppermint oil, oil of

wintergreen, cherry or orange flavorings, colorings, stabilizers such as methyl- or propyl-paraben in order to obtain biologically acceptable preparations.

The choice of vehicle and the content of the active substance in the vehicle are generally determined in accordance with the solubility and chemical properties of that product, the particular mode of administration, and the provisions to be observed in preparing the form of administration. For example, excipients such as lactose, sodium citrate, calcium carbonate, dicalcium phosphate, and disintegrating agents such as starch, alginic acids, and certain complex silica gels combined with lubricants such as magnesium stearate, sodium lauryl sulfate, and talc may be used for preparing tablets, troches, pills, capsules, and the like. To prepare a capsule, it is advantageous to use lactose and liquid carriers, such as high molecular weight polyethylene glycols. Various other materials may be present as coatings or to otherwise modify the physical form of the dosage unit. For instance, tablets, pills, or capsules may be coated with shellac, sugar, or both. When suspensions are used they may contain emulsifying agents or agent which facilitate suspension. Diluents such as sucrose, ethanol, polyols, such as polyethylene glycol, propylene glycol, and glycerol, and choloform or mixtures thereof may also be used. In addition, the divalent metal salt may be incorporated into sustained-released preparations and formulations.

For oral administration, the divalent metal salt may be administered, for example, with an inert or diluent or with an assimilable edible carrier, or it may be enclosed in hard or soft shell gelatin capsules, or it may be compressed into tablets, or it may be incorporated directly with the food of the diet, or may be incorporated with excipient and used in the form of ingestible tablets, buccal tablets, troches, capsules, elixirs, suspensions, syrups, wafers, and the like.

For parenteral administration, emulsions or suspensions of the divalent metal salt in a vegetable oil, for example sesame oil, groundnut oil or olive oil, or aqueous-organic solutions such as water and propylene glycol, as well as injectable organic esters such as ethyl oleate are used. The injectable forms must be fluid to the extent that it can be easily syringed and proper fluidity maintained, for example, by the use of a coating such as lecithin, by the maintenance of the required particle size in the case of dispersions, and by the use of surfactants. Prolonged absorption of the injectable compositions can be brought about by the use of agents delaying absorption, for example, aluminum monostearate and gelatin. Dispersions can also be prepared in glycerol, liquid polyethylene glycols and mixtures thereof, and in oils. Generally, injectable dispersions are prepared by incorporating the various sterilized active ingredients into a sterile vehicle which contains the basic dispersion medium and the required other ingredients form those enumerated above.

For topical administration, gels (water or alcohol based), creams, or ointments containing compounds of the invention may be used. Such formulations are essentially conventional and include cosmetic formulations for the skin, hair, nails, and the like.

The fatty acid divalent metal salts of the present invention function as rumen bypass feed supplements and may be conveniently fed to a ruminant admixed with a conventional ruminant feed. The feeds are typically vegetable materials edible by ruminants, such as legume hay, grass hay, corn silage, grass silage, legume silage, corn grain, oats, barley, distiller's grain, brewer's grain, soy bean meal and cottonseed meal and are included in an amount as typically recommended by a husbandry nutritionist, which ordinarily does ont exceed 5% by weight of the dry solids content of the feed.

The divalent metal salts are also useful in general as nutritional supplements for humans, other mammals, and non-mammals, including birds and fish. Thus, methods in accordance with the present invention add an effective amount of the beneficial unsaturated fatty acid divalent metal salts of the present invention to a food product including pet food products such as cat food and dog food. Effective amounts include amounts that will provide a food product having a beneficial unsaturated fatty acid content between about 0.05 and about 1.5 weight percent. A beneficial unsaturated fatty acid content between about 0.1 weight percent and about 0.5 weight percent is preferred. Among the beneficial unsaturated fatty acids, polyunsaturated fatty acids, including conjugated polyunsaturated fatty acids, are preferred. This would include non-conjugated omega-3 and omega-6 fatty acids and conjugated fatty acids such as CLA's. Among the omega-3 fatty acids DHA, EPA, DPA and LNA are preferred. Among the omega-6 fatty acids, linoleic acids and arachidonic acids are preferred.

The present invention also includes food products containing the beneficial unsaturated fatty acid divalent metal salts of the present invention within the ranges described, including pet food products such as cat food and dog food. The cat foods and dog foods include dry, semi-moist and moist cat food and dog food prepared by otherwise conventional methods from conventional formulations incorporating conventional pet food ingredients to which the divalent metal salts are added by techniques conventionally employed for the nutritional supplementation of these products. The divalent metal salts may be blended with the pet food components or, in the case of extruded dry and semi-dry products, sprayed or dusted on the surface thereof, with or without components such as palatability enhancers.

The omega-3 fatty acid divalent metal salts of the present invention, and particularly those containing one or more omega-3 fatty acids selected from DHA, EPA, DPA and LNA can be used in the fertility enhancement methods disclosed by U.S. Patent No. 6,576,667, which is incorporated herein by reference. Applicants have since learned that in addition to DHA and EPA, DPA and LNA also enhance the fertility of ruminants and other animals. The present invention makes possible the preparation of fatty acid divalent metal salts with higher levels of omega-3 fatty acids, thereby reducing the quantities that must be fed to obtain the beneficial effect. The present invention therefore includes methods according to that patent using the omega-3 fatty acid divalent metal salts of the present invention, including the additionally disclosed omega-3 fatty acid divalent metal salts not mentioned in that patent.

This should not be interpreted as limiting the scope of the present invention, which provides a means by which beneficial unsaturated fatty acid rich divalent metal salts may be prepared having utility as nutritional supplements for essentially any animal for which omega-3 fatty acids provide nutritional or therapeutic benefit.

The following non-limiting examples set forth herein below illustrate certain aspects of the invention. All parts and percentages are by weight unless otherwise noted, and all temperatures are in degrees Celsius.

EXAMPLES

Example 1:

In this example, 300 grams of soy oil was charged to a vessel equipped with mixing blades. A nitrogen blanket was applied to the vessel. The soy oil was then heated to 50°C. After the oil reached 50°C , 45 grams of magnesium hydroxide was added and thoroughly mixed. The vessel was then resealed. The mixture was stirred and heated until the temperature rose to 285°C, at which point the saponification reaction occurred and the temperature rose to 290°C. The reacted material was removed from the vessel and placed in flat pan for cooling. After cooling, the material processed into small granules of a dry, free-flowing magnesium salt.

What is claimed is:

1. A method for the preparation of a free-flowing, storage-stable fatty acid divalent metal salt product comprising:

forming a reactive admixture comprising (a) an unsaturated fatty acid glyceride feedstock; and (b) from about 10% to about 30% of the total admixture weight of at least one divalent metal hydroxide; and

heating the admixture to a temperature at which said fatty acid glycerides saponify to form fatty acid divalent metal salts in an atmosphere in which the partial pressure of oxygen has been reduced by an amount effective to provide an improvement in storage stability until a free-flowing, storage-stable product is obtained;

wherein said divalent metal is selected from the group consisting of calcium, copper, iron, magnesium, manganese, selenium, and zinc.

2. The method of claim 1, wherein said partial pressure of oxygen is reduced by inert gas blanketing of said admixture while heating.

3. The method of claim 2, wherein said inert gas comprises nitrogen.

4. The method of claim 1, wherein said partial pressure of oxygen is reduced by heating said admixture under vacuum.

5. The method of claim 1, wherein said unsaturated fatty acid glyceride feedstock comprises an unsaturated fatty acid concentration sufficient to form unstable divalent metal salt products when saponified in an ambient atmosphere

6. The method of claim 1, wherein said unsaturated fatty acid glyceride feedstock comprises polyunsaturated fatty acids.

7. The method of claim 6, wherein said polyunsaturated fatty acids are selected from the group consisting of omega-3 and omega-6 fatty acids and combinations of either or both.

8. The method of claim 7, wherein said polyunsaturated fatty acids comprise one or more omega-3 fatty acids selected from the group consisting of DHA, EPA, DPA and LNA.

9. The method of claim 6, wherein said polyunsaturated fatty acids comprise one or more conjugated fatty acids.

10. The method of claim 9, wherein said one or more conjugated fatty acids comprise one or more CLA isomers.

11. The method of claim 1, wherein said fatty acid glyceride feedstock comprises a mixture of two or more C_{10}-C_{22} fatty acids having greater than about 45% by weight of the fatty acid content in the form of fatty acid glycerides.

12. The method of claim 11, wherein about 85 and about 100% by weight of said fatty acid mixture is in the form of fatty acid glycerides.

13. The method of claim 1, wherein said fatty acid glyceride feedstock comprises from about 40 to about 95 % by weight of unsaturated fatty acids.

14. The method of claim 1, wherein said feedstock comprises up to about 100 % by weight of marine oil.

15. The method of claim 14, wherein said marine oil is selected from the group consisting of menhaden, herring, mackerel, caplin, tilapia, tuna, sardine, pacific saury, krill, kelp, and algae oils.

16. The method of claim 15, wherein said marine oil comprises one or more omega-3 or omega-6 fatty acids selected from the group consisting of DHA, EPA, DPA, LNA, linoleic acid and arachidonic acid.

17. The method of claim 1, further comprising the step of cooling said admixture and forming a solid, free-flowing and granular fatty acid divalent metal salt product.

18. The method of claim 17, wherein said admixture is cooled in said atmosphere in which said partial pressure of oxygen has been reduced by an amount effective to provide an improvement in storage stability.

19. The method of claim 1, wherein said heating step comprises preheating said fatty acid feed stock to a temperature from about 350°F to about 550°F and said forming step comprises rapidly forming a uniform homogeneous blend of said fatty acid feedstock and said divalent metal hydroxide at a rate effective to produce an improvement in storage stability in said divalent metal salt product.

20. The method of claim 1, wherein said divalent metal hydroxide is formed in said reactive admixture by combining stoichiometric quantities of corresponding divalent metal oxide and water.

21. A fatty acid divalent metal salt prepared by the method of claim 1, wherein said divalent metal salt comprises one or more beneficial unsaturated fatty acids.

22. A fatty acid divalent metal salt according to claim 21, comprising polyunsaturated fatty acids.

23. The fatty acid divalent metal salt of claim 22, wherein said polyunsaturated fatty acids are selected from the group consisting of omega-3 and omega-6 fatty acids and combinations of either or both.

24. The fatty acid divalent metal salt of claim 23, wherein said polyunsaturated fatty acids comprise one or more omega-3 or omega-6 fatty acids selected from the group consisting of DHA, EPA, DPA, LNA, linoleic acid and arachidonic acid..

25. The fatty acid divalent metal salt of claim 24, comprising at least one polyunsaturated fatty acid selected from the group consisting of about 1 to about 25% by weight DHA, about 1 to about 25% by weight EPA, about 1 to about 25% by weight DPA, about 1 to about 75% by weight LNA, about 0.5 to about 10 % by weight arachidonic acid, about 1 to about 80% by weight linoleic acid and about 1 to about 80% by weight CLA.

26. The fatty acid divalent metal salt of claim 22, wherein said polyunsaturated fatty acids comprise one or more conjugated fatty acids.

27. The fatty acid divalent metal salt of claim 26, wherein said one or more conjugated fatty acids comprise one or more CLA isomers.

28. A fatty acid divalent metal salt prepared by the method of claim 14.

29. A fatty acid divalent metal salt prepared by the method of claim 15.

30. A fatty acid divalent metal salt prepared by the method of claim 16.

31. The fatty acid divalent metal salt of claim 30, wherein said marine oil is selected from the group consisting of menhaden, herring, mackerel, caplin, tilapia, tuna, sardine, pacific saury and krill oils.

32. The fatty acid divalent metal salt of claim 30, wherein said marine oil comprises one or more omega-3 or omega-6 fatty acids selected from the group consisting of DHA, EPA, DPA, LNA, linoleic acid and arachidonic acid.

33. A storage-stable divalent metal salt saponification product of an unsaturated oil consisting essentially of one or more marine oils.

34. The saponification product of claim 33, wherein said one or more marine oils are selected from the group consisting of menhaden, herring, mackerel, caplin, tilapia, tuna, sardine, pacific saury and krill oils.

35. The saponification product of claim 33, wherein said one or more marine oils comprise one or more fatty acids selected from the group consisting of omega-3 and omega-6 fatty acids.

36. The saponification product of claim 35, wherein said one or more marine oils comprise one or more omega-3 or omega-6 fatty acids selected from the group consisting of DHA, EPA, DPA, LNA, linoleic acid and arachidonic acid.

37. The saponification product of claim 36, comprising at least one polyunsaturated fatty acid selected from the group consisting of about 1 to about 25% by weight DHA, about 1 to about 25% by weight EPA, about 1 to about 25% by weight DPA, about 1 to about 75% by weight LNA, about 0.5 to about 10 % by weight arachidonic acid, about 1 to about 80% by weight linoleic acid and about 1 to about 80% by weight CLA.

38. A storage-stable fatty acid divalent metal salt saponification product of a fatty acid glyceride feedstock having an unsaturated fatty acid concentration sufficient to form unstable divalent metal salt products when saponified in an ambient atmosphere.

39. The fatty acid divalent metal salt of claim 38, wherein said unsaturated fatty acid glyceride feedstock comprises polyunsaturated fatty acids.

40. The fatty acid divalent metal salt of claim 39, wherein said polyunsaturated fatty acids are selected from the group consisting of omega-3 and omega-6 fatty acids and combinations of either or both.

41. The fatty acid divalent metal salt of claim 40, wherein said polyunsaturated fatty acids comprise one or more omega-3 or omega-6 fatty acids selected from the group consisting of DHA, EPA, DPA, LNA, linoleic acid and arachidonic acid.

42. The fatty acid divalent metal salt of claim 41, comprising at least one polyunsaturated fatty acid selected from the group consisting of about 1 to about 25% by weight DHA, about 1 to about 25% by weight EPA, about 1 to about 25% by weight DPA, about 1 to about 75% by weight LNA, about 0.5 to about 10% by weight arachidonic acid, about 1 to about 80% by weight linoleic acid and about 1 to about 80% by weight CLA.

43. The fatty acid divalent metal salt of claim 39, wherein said polyunsaturated fatty acids comprise one or more conjugated fatty acids.

44. The fatty acid divalent metal salt of claim 43, wherein said one or more conjugated fatty acids comprise one or more CLA isomers.

45. The fatty acid divalent metal salt of claim 38, wherein said fatty acid glyceride feedstock comprises from about 50 to about 85% by weight of unsaturated fatty acids.

46. A method for increasing fertility in an animal, comprising feeding an animal in need thereof an effective amount of a composition of Claim 41 comprising at least DHA or EPA.

47. The method of Claim 46, wherein said animal is a male or female ruminant.

48. The method of Claim 47, wherein said ruminant is a dairy cow.

49. The method of Claim 48, comprising starting the feeding of said product to said ruminant between about 21 days before and about 28 days after parturition.

50. The method of Claim 49, wherein said feeding of said product to said ruminant is continued at least until conception occurs.

51. The method of Claim 47, wherein said product is fed to said ruminant daily.

52. The method of Claim 46, wherein said product is fed to said animal for at least 30 days after conception.

53. The method of Claim 52, wherein said product is fed to said animal for at least 60 days after conception.

54. The method of Claim 53, wherein said product is fed to said animal for at least 150 days after conception.

55. The method of Claim 50, wherein the feeding of said product is discontinued at conception or within 150 days thereafter and said method further includes the step of feeding daily to said ruminant a second fatty acid calcium salt product for supplying milk production energy to a female ruminant after the feeding of the first product is discontinued.

56. A nutritional supplement composition comprising at least one fatty acid divalent metal salt according to claim 21, 34, or 38 and a biologically acceptable carrier.

57. The method of claim 1, wherein said reactive admixture comprises a plurality of said divalent metal hydroxides.

58. The fatty acid divalent metal salt product of claim 21, 34, or 38 comprising a plurality of divalent metals.

59. A food product for a companion animal comprising the fatty acid metal salt product of claim 21, 34, or 38.

ABSTRACT

Methods for the preparation of a free-flowing, storage-stable fatty acid divalent metal salt product by forming a reactive admixture comprising (a) an unsaturated fatty acid glyceride feedstock; and (b) from about 10% to about 30% of the total admixture weight of at least one divalent metal hydroxide; and heating the admixture to a temperature at which said fatty acid glycerides saponify to form fatty acid divalent metal salts in an atmosphere in which the partial pressure of oxygen has been reduced by an amount effective to provide an improvement in storage stability until a free-flowing, storage-stable product is obtained; wherein said divalent metal is selected from the group consisting of calcium, copper, iron, magnesium, manganese, selenium, and zinc. Storage stable divalent metal salts of unsaturated fatty acids prepared by the inventive method are also disclosed.

M:\JJohnson\Bioproducts\P-22,888F USA\Provisional Application.doc

FIG. 1

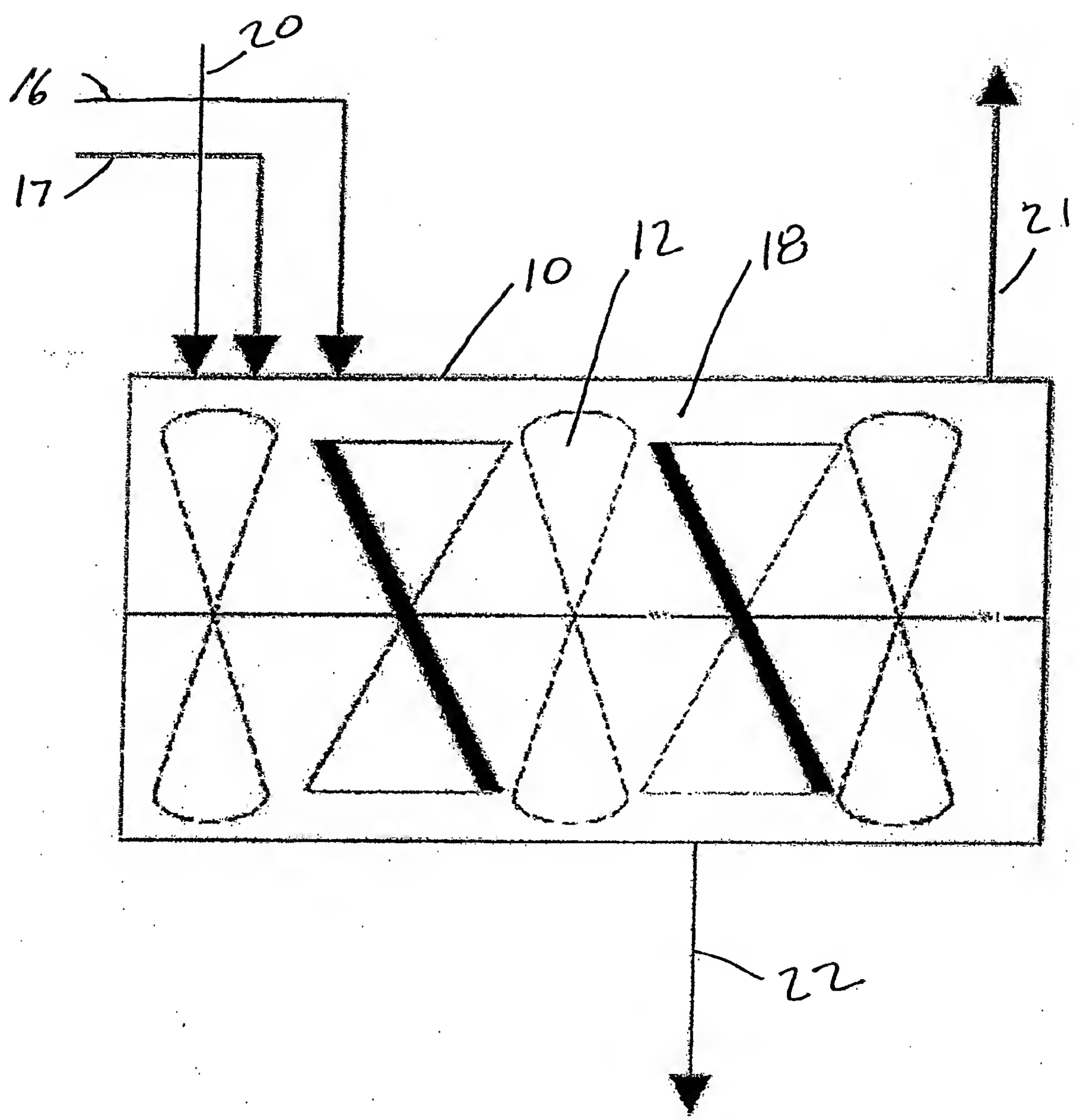
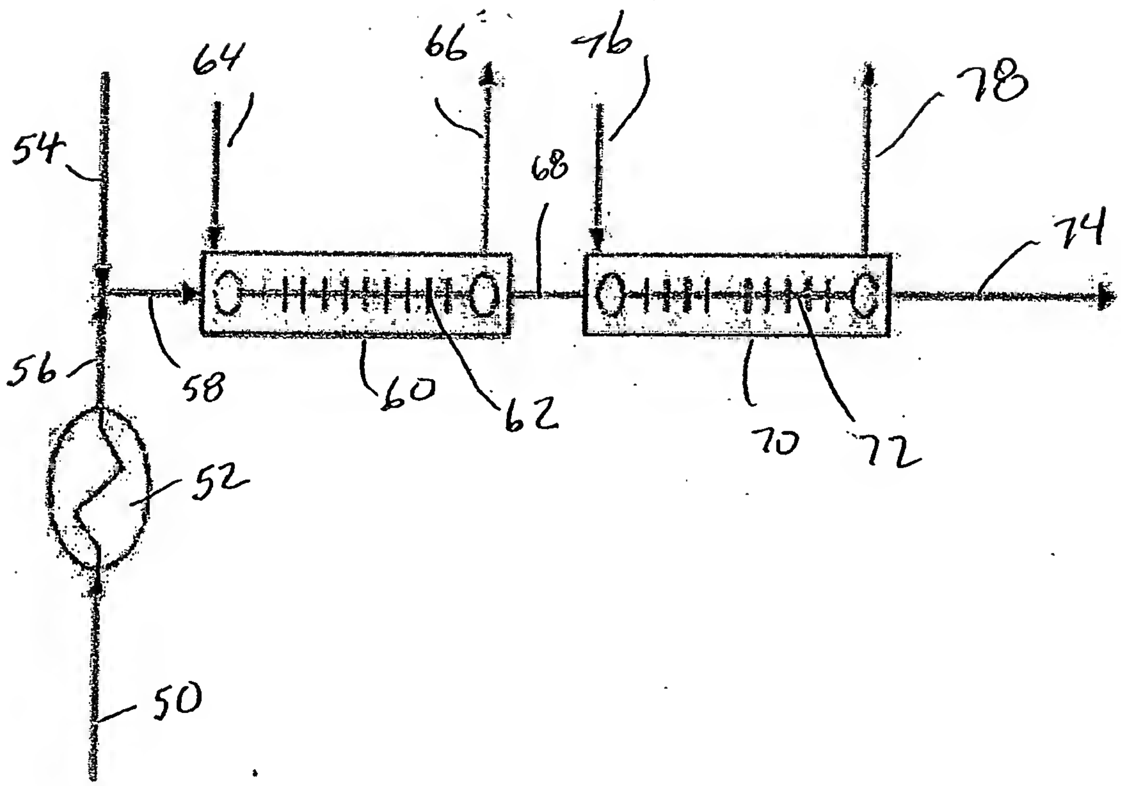


FIG. 2



Patent

Express Mail Label No. EV143409679US
Attorney Docket No. P22,888-F USA

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of:
George K. Strohmaier
Nestor Daniel Luchini
Eiler D. Frederiksen
Bob J. Dull

Application No.: **N/A**

Filing Date: **HEREWITH**

For: **POLYUNSATURATED FATTY ACID DIVALENT METAL SALT SYNTHESIS**

Group Art Unit: **N/A**

Examiner: **N/A**

Mail Stop Provisional Application
Commissioner for Patents
P. O. Box 1450
Alexandria, VA 22313-1450

APPLICATION DATA SHEET
37 C.F.R. § 1.76

Application information

Application Type::	Provisional
Subject Matter::	Utility
Suggested Classification::	
Suggested Group Art Unit::	
CD-ROM or CD-R?::	None
Title::	Polyunsaturated Fatty Acid Divalent Metal Salt Synthesis
Attorney Docket Number::	P22,888-F USA
Request for Early Publication?::	No
Request for Non-Publication?::	No
Suggested Drawing Figure::	

Total Drawing Sheets::	Two (2)
Small Entity::	No
Petition included?::	No
Secrecy Order in Parent Appl.?::	No

Applicant information

Applicant Authority Type::	Inventor
Primary Citizenship Country::	USA
Status::	Full Capacity
Given name::	George
Middle Name::	K.
Family Name::	Strohmaier
City of Residence::	Medina
State or Province of Residence::	Ohio
Country of Residence::	United States of America
Street of mailing address::	1111 Ty Drive
City of mailing address::	Medina
Country of Mailing Address::	United States of America
State or Province of mailing address::	Ohio
Postal or Zip Code of mailing address::	44526

Applicant Authority Type::	Inventor
Primary Citizenship Country::	USA
Status::	Full Capacity
Given name::	Nestor
Middle Name::	Daniel
Family Name::	Luchini
City of Residence::	Naperville
State or Province of Residence::	Illinois
Country of Residence::	United States of America

Street of mailing address::	1469 Culpepper Drive
City of mailing address::	Naperville
Country of Mailing Address::	United States of America
State or Province of mailing address::	Illinois
Postal or Zip Code of mailing address::	60540

Applicant Authority Type::	Inventor
Primary Citizenship Country::	USA
Status::	Full Capacity
Given name::	Eiler
Middle Name::	D.
Family Name::	Frederiksen
City of Residence::	Henderson
State or Province of Residence::	Nevada
Country of Residence::	United States of America
Street of mailing address::	1140 Calico Ridge
City of mailing address::	Henderson
Country of Mailing Address::	United States of America
State or Province of mailing address::	Nevada
Postal or Zip Code of mailing address::	89015

Applicant Authority Type::	Inventor
Primary Citizenship Country::	USA
Status::	Full Capacity
Given name::	Bob
Middle Name::	J.
Family Name::	Dull
City of Residence::	O'Fallon
State or Province of Residence::	Illinois
Country of Residence::	United States of America

Street of mailing address::	722 Victoria Lane
City of mailing address::	O'Fallon
Country of Mailing Address::	United States of America
State or Province of mailing address::	Illinois
Postal or Zip Code of mailing address::	62269

Correspondence Information

Correspondence Customer Number:: 23307

Representative Information

Representative Customer Number::	23307

Signature of Representative

April 30, 2004
Date

Jimmie Johnson

Jimmie D. Johnson, Reg. No. 52,485
SYNNESTVEDT & LECHNER LLP
2600 Aramark Tower
1101 Market Street
Philadelphia, PA 19107-2950
Telephone (215) 923-4466
Facsimile (215) 923-2189

PJB:bgm

M:\PButch\Bioproducts\P22888-F USA\PatentOffice\ApplicationDataSheet.wpd